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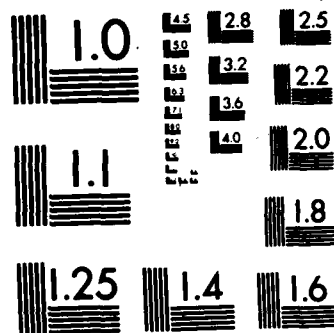
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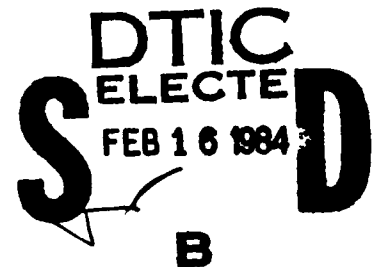
Vibrational Energy Transfer at a Surface in Reacting
Systems. Decomposition of Azomethane.

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Azomethane Decomposition

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E sub 0 approx. 50 kcal/mole

Abstract

Single collision activation of the "energetic" species azomethane at a hot seasoned fused silica surface was studied. Excitation above the critical threshold ($E_0 \approx 50 \text{ kcal mole}^{-1}$) was monitored by subsequent homogeneous decomposition. The collisional efficiency was studied over the range 700K to 875K. Energy transfer efficiency is discussed and compared with values for other substrate molecules of comparable size and polarity. The data emphasize the uncertainty that still prevails with respect to the values of the Arrhenius parameters for this system.



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Introduction

In continuation of studies on single-collision vibrational excitation of molecules at a seasoned fused silica surface [1-3], we have examined the molecule azomethane. Azomethane is potentially explosive and is an example of an "energetic" molecule. The reaction threshold ($E_0 \approx 50 \text{ kcal mole}^{-1}$) constitutes an absorbing barrier and the flux of molecules scaling the barrier may be monitored by disappearance of substrate molecules. Other single-collision systems previously studied were cyclobutene [1,2] and methyl cyclobutene [3] which have reaction thresholds just above $30 \text{ kcal mole}^{-1}$. The higher threshold for azomethane is coupled with a higher Arrhenius frequency factor [4,5]. This ensures both that the specific decomposition probabilities $k(E)$ are sufficiently large near threshold, so that measurable decomposition takes place in the temperature region studied and, also, that the system is close to the second order region so that virtually all molecules that are excited above threshold decompose under the particular conditions of pressure and of free flight times employed. This is a desirable condition of study for the present purpose since it eliminates the need for specification of a transition state as well as any error in modeling $k(E)$, because no stabilization correction, following excitation, is necessary.

Before collision with the hot surface, molecules are first equilibrated at a lower temperature. Molecules that are insufficiently excited by collision and do not react are re-equilibrated thereafter at the lower temperature by repeated collisions with the cold surface. The population vector after one collision is given by $N^1 = PN^0$, where N^0 is the original (cold) population distribution. It is to our purpose to gain information about the collisional transition probability matrix P .

The hot surface employed in this study, fused silica, is a "dirty" one from the point of view of ultra high vacuum surface studies. It is a "seasoned" one from the point of view of conventional thermal unimolecular decomposition studies, i.e. the hot surface is first cleaned by oxidation and is then treated with repeated small doses of substrate until a lowest rate is obtained that, hopefully, signifies that no surface catalysis occurs. In this system, the data indicate that heterogeneous reaction may be of importance, especially at lower temperatures, but the situation is clouded by existing uncertainty in the Arrhenius activation energy for the reaction.

Experimental

Materials

Trans-Azomethane [6] was prepared according to the method of Jahn [7] and Diels and Koll [8] from dimethylhydrazine dihydrochloride (Aldrich). To avoid possible decomposition on a glpc column, the azomethane was purified by condensation chromatography using a 20 cm x 1 cm copper powder column at -30°C and $+18^{\circ}\text{C}$ with FID. The material was analyzed by mass spectrometry; no interfering impurities were found.

Apparatus and Procedure

The reaction mixture contained equal amounts of azomethane with cyclopropane as an internal analytical standard. The reaction system apparatus used is a modification of that employed in the variable encounter method (VEM) and was the same as that used for previous single collision experiments [1-3]. The reaction vessel consisted of an ice-cooled 3-l pyrex flask fitted with a central tubular (3.5 cm o.d.) fused quartz finger. The finger carried a metal bath in its interior having heater, stirrer and thermocouple well. The finger reactor temperature was varied from 700K to 900K at constant wall temperature of 273K.

Before measurements were made, the silica surface was "seasoned" at the highest temperature by exposure to the reaction mixture at a pressure of 10^{-3} - 10^{-2} torr until a lowest rate was achieved. The seasoning was maintained by occasional exposure of the system for a few hours to the mixture gas at a pressure of 1 - 5×10^{-3} torr, which was then pumped to a residual pressure of $\sim 10^{-6}$ torr before a run. The system was run in static mode in the substrate pressure region of 1 - 3×10^{-4} torr with residence times ranging from 15 min to 5 hours. Pressure measurements were made with an

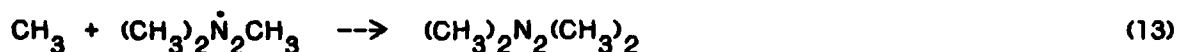
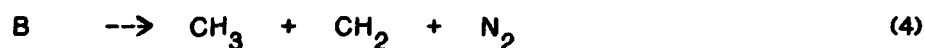
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Analysis was performed by gas chromatography on 150 ft. SCOT squalane column at 0°C with FID detection.

Results and Discussion

The reaction probability per collision P_c was calculated from the relation [1-3], $P_c = 4V\bar{c}S$, where V is the volume of the reactor (2850 cm^3); S is the surface area of the finger (78 cm^2); k is the measured first order rate constant (sec^{-1}); \bar{c} is the average speed of the cold molecule, $\bar{c} = (8k_B T_0 / \pi m)^{1/2}$, where T_0 is the flask temperature; $\bar{c} = 1.92 \times 10^3 T_0^{1/2}$; then, $P_c = 0.0761 \text{ k/T}_c^{1/2}$.

In addition to the uncertainties attaching to this system and mentioned earlier, the reaction is known to involve chain character and possible catalytic complications. We will discuss each of these. Paquin and Forst [19] have employed a mechanism, some principal steps of which are as follows:



B is considered to display allylic type resonance. Paquin and Forst have given expressions for the chain yield, L , in terms of side product amounts. Over a narrow range of temperatures around 600K and over a wide range of pressures from 20 to 400 torr they found that L increases with temperature increase and pressure decrease. Values of L in their work varied in the range 2.2 to 2.8. For our work, at higher temperature and lower pressure, we have adopted $L=3$. We believe that this is probably a reasonable upper limit.

Very recently, Marshall and Shahkar [10] have investigated the effect of surface complications in Pyrex vessels. They found no significant dependence of the measured rates on surfaces treated in different ways -- whether acid-washed, alkali-washed or salt(KCl)solution-washed. The data were reproducible ($\pm 8\%$). Catalytic reaction was concluded to be a minor (2%) molecular decomposition process that gives C_2H_6 split-off. Even more important, they concluded that the Arrhenius parameters for reaction (1) are $\log A = 13.64$, $E_a = 45.6 \text{ kcal mole}^{-1}$, which agreed well with their earlier work and a value cited by them due to Lin and Laidler. They adopted $\log A = 13.8$ and $E_a = 46.0 \text{ kcal mole}^{-1}$ as a grand average. This value of E_a is at least 3 kcal mole^{-1} smaller than that usually cited for the initial process. This uncertainty, newly appeared relative to the time the present work was nearing completion, makes difficult our ability to derive definitive quantitative conclusions from the present work.

Average values of P_c are summarized in Tabel I. The value at each temperature is the mean of 3-5 separate runs. The data are plotted in Fig. 1 together with the theoretical strong collider curves using $E_0 = 49.4$ and $46.0 \text{ kcal mole}^{-1}$. The former value was calculated on the basis of an average of Arrhenius parameters cited in refs. 4 and 5; the latter value is derived from ref. 10. Also given in Table I for 800K and 875K is $\langle \Delta E_d \rangle$, the average value of the down jump step calculated on the basis of gaussian and Boltzmann-exponential forms [2,3] for the analytical representation of the elements p_{ij} of P . Two aspects are evident from Fig. 1 and the values given in Table I. First, the values of $\langle \Delta E_d \rangle$ are ~30-100% higher than those found for cyclobutene at comparable temperatures (800K) and listed in ref. 11. Second, the experimental curve approaches very close to the strong collider curve

below 700K, on the $E_0=49\text{kcal}$ basis; this may be evidence for some catalytic effect of the surface which, as pointed out much earlier in connection with VLPP work [12], becomes more prominent at lower temperatures where reaction rate and P_c decline because of the conventional behavior of the Boltzmann population distribution. Both of the above findings depend on the reliability of the experimental Arrhenius activation energy employed. Use of the lower value moves the strong collider reference curve up and decreases the values of $\langle\Delta E_d\rangle$. It is known that the cyclobutane and cyclobutene systems are highly reproducible and essentially free of surface catalysis complications. There is no real reason from the rate data to believe that energy transfer is unusually efficient for azomethane and this would support low values of E_a such as ref. 10. Relative to the results on polar iodopropane [13], for example, the data for azomethane suggest only comparable efficiency.

Acknowledgment

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Table I. Experimental Values of P_c and $\langle \Delta E_d \rangle$ for Azomethane

T/K	700	750	800	850	875
$P_c (\times 10^8)$	$1.1 \pm 0.17(5)^a$	$4.2 \pm 1.0(3)$	$15 \pm 2(3)$	$74 \pm 2.7(3)$	$97 \pm 7(5)$
$\langle \Delta E_d \rangle / \text{cm}^{-1} \text{ }^b$			8150 (G.E = 49.4) 9950 (BE, $E_0 = 49.4$)		7600 (G.E = 49.4) 8600 (BE, $E_0 = 49.4$)
			6250 (G.E = 46.0) 5500 (G.E $_0 = 46.0$) ^c 4330 (G.ref $_0$ \square)		
			6950 (BE, E_0 \square) 4230 (BE, E_0 \square)		

^a Based on $L=3$. Standard deviation of the mean of the number of measurements shown in parentheses.

^b Calculated at $E=E_0$.

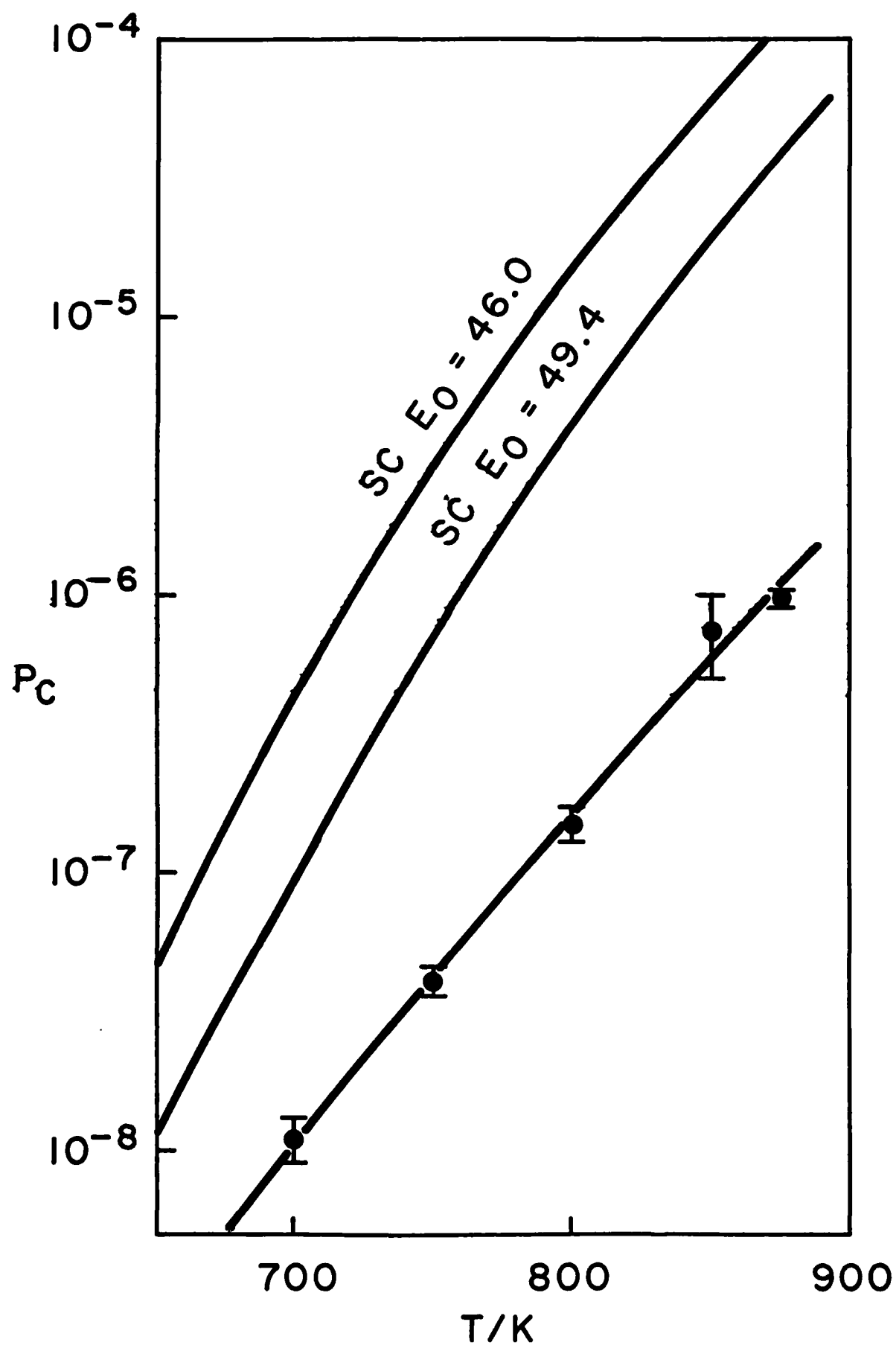
^c Calculated for a libration model for the hindered internal rotors (2).

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Captions

Fig. 1. Plot of P_c (log scale) vs. temperature (K) for azomethane single collisions on fused silica. The strong collider theoretical curves (SC) are based on critical thresholds of 49.4 and 46.0 kcal mole⁻¹.



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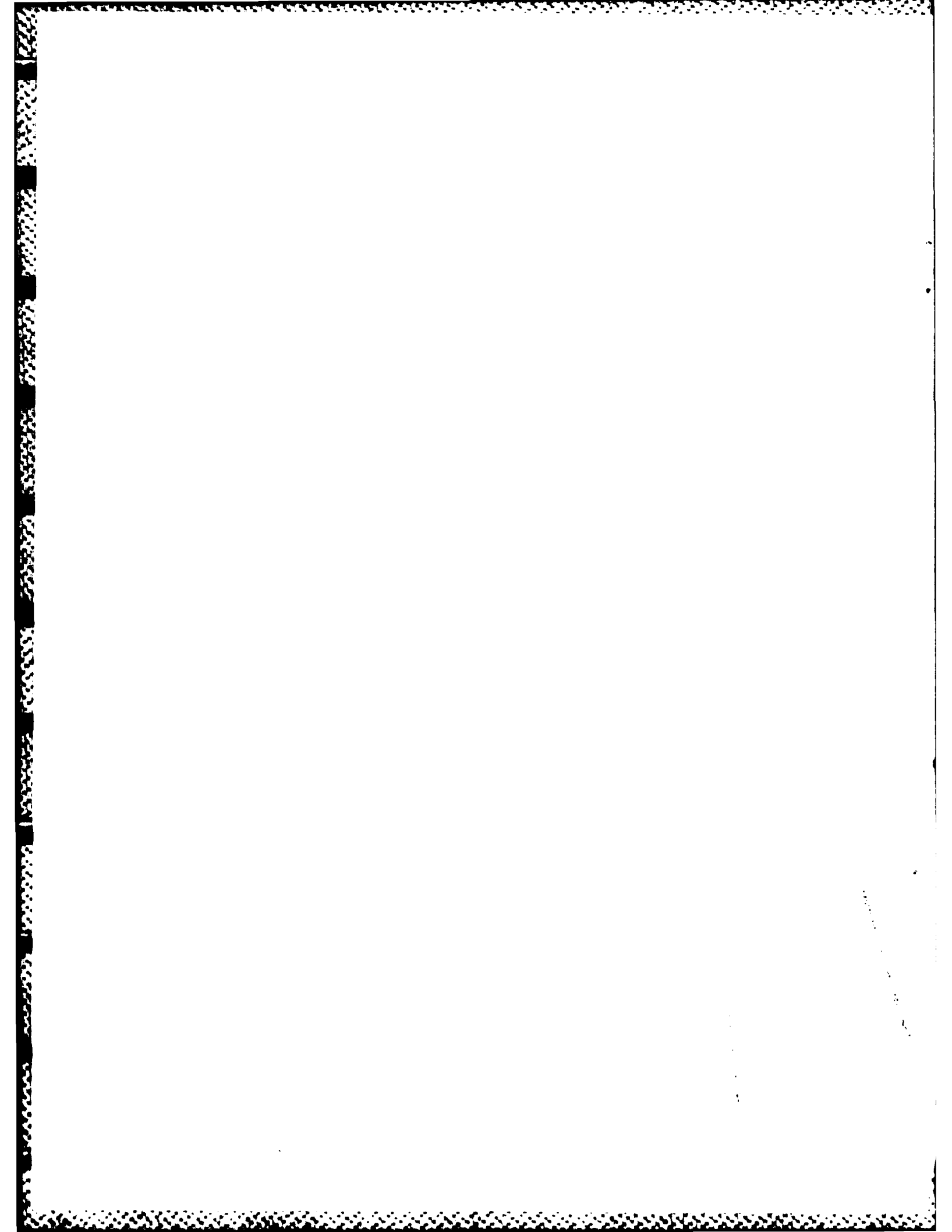
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